

TITLE

Infiltrated Cobalt Attachment Ring for A CBN Sintered Body and Its Fabrication Process

CROSS-REFERENCE TO RELATED APPLICATIONS

None

FEDERAL SPONSERSHIP

None

BACKGROUND OF THE INVENTION

Field of the invention

This invention relates to cubic boron nitride sintered bodies used as cutting tools

Description of related art

Super abrasive materials are useful today for cutting metals and creating heat sinks, wire dyes, drill bits, blades, and inserts. Diamond is utilized in many of these functions, but because diamond is highly reactive with iron under high temperatures, cutting steel or other ferrous metals will destroy diamond cutters. Cubic boron nitride, the second hardest material, is favorable in cutting ferrous material because it is much more stable than diamond under these conditions.

Sintering is known as the process by which diamond or boron cubic nitride grains are exposed to high temperature and high pressure in order to bind them together and make useful bodies of these materials. High enough temperatures combined with high enough pressures reduce the surface areas of the grains by essentially fusing the grains together and the grains form intercrystalline bonds between one another. Cubic boron nitride is inert by itself due to a high amount of oxides bound to the surface of the grains. To overcome the inert nature of the cubic boron nitride, adjuvant material is mixed in

with the CBN grains to act as catalysts in the bond formations. The adjuvant material is commonly metals such as aluminum, titanium, cobalt, nickel, or silicon. Cubic boron nitride and diamond sintered bodies are often bonded to a carbide substrate. One of the most common substrates is a tungsten carbide with a concentration of cobalt that actually melts into the CBN or diamond preform and helps catalyze bonds during sintering.

While the metal binders are useful in the sintered bodies, they do create some problems. Binders that are near the surface of the sintered body are exposed to high amounts of heat which cause the metals to expand. Since the metal binders don't expand at the same rate as the CBN or the diamond, a stress is created within the sintered body which can lead to cracking the sintered body. Attempts to resolve this problem deal with trying to use binders that have the same thermal expansion rate as the diamond or CBN. Others attempts have shown that by purging several millimeters of the surface of the sintered body of the binders actually increases its wear resistance. Purging the binders too deep however, will cause the sintered body to be more brittle. This invention tries to reduce this thermal expansion problem, by adding binders of a smaller size, so that the individual expansion of each grain causes less stress on the sintered body.

Another issue during sintering is the amount of oxides bound to the surface of the CBN or diamond grains. On diamond, oxide complexes of $C=O$, $C-O-O-H$, $C-O-H$, $C-O-Fe(3+)$, $C-O-Al(2+)$ are formed. Cubic boron nitride has similar oxide complexes that bind to the boron. These boron oxides occupy binding sites that could be used to bind to other CBN grains. Preparing the preforms in a non-oxidizing environment and using carbide and aluminum binders are current methods used to reduce the amount of oxides on the surface of the CBN or diamond grains. In our preferred embodiment, we use

titanium and aluminum in preparing our preform. These metals are also subject to oxidation which would also interfere with binding. In our preferred embodiment, diamond is used effectively as a reducing agent, which reduces the CBN, titanium, and aluminum grains and allow for more and stronger bonds between grains.

Another important feature that CBN sintered bodies need is adequate attachment to its substrate. Weak bonds will result in cracking of both materials due to low impact and wear resistances. In our present invention, we use infiltrated cobalt to form a unique attachment ring.

BRIEF SUMMARY OF THE INVENTION

The present invention improves upon existing sintered CBN bodies by providing not only a strong attachment to its tungsten carbide substrate, but also by improving bond strength through smaller grain sizes and providing more opportunities between CBN grains because of a reduction reaction included in the process of making the preform.

The cobalt attachment ring provides a stronger hold of the sintered body to the tungsten carbide substrate. While cobalt is found throughout the entire CBN sintered body, it is concentrated near its edge. The width of the infiltrated cobalt ring spans between 15 microns to 30 microns from the edge of the sintered body, being wider at the corners. If a strong attachment of the CBN sintered body, to the tungsten carbide, is not formed then wear resistance decreases and the life span of the CBN sintered body also decreases.

Another unique feature of the present invention is the grain size throughout the

sintered body. The aluminum and titanium are obtained by adding the CBN, diamond, and aluminum nitride grains into an aluminum sleeve with titanium balls. For 24 hours the titanium balls mill in the aluminum sleeve breaking off submicron particles of titanium and aluminum which mix into the mixture. The CBN grains in our preferred embodiment range from 1 to 2 microns, the AlN grains range from .3 to .5 microns, and the diamond grains range from .5 to 2.5 microns. The smaller the grains size of the particles, the greater the surface area in the mixture. This allows for more bonding between molecules which results in greater wear resistance. Another function of the smaller grain sizes is reducing the surface friction of the CBN sintered body. Larger grain sizes will provide an uneven surface; for example, if the average grains size of the sintered compact are 20 microns then the surface will be rougher than the surface when the grain sizes were only .5 microns. Lower friction on the surface will allow for more efficient use of energy from the cutting tools and less strain on the sintered body as well. A smoother edge will concentrate the force directly under the cutter's edge and not throughout extra material.

The removal of the oxides bound to the CBN grains will allow for more bonding between the CBN grains and strengthen the CBN bonds that currently exist. The increased number of bonds and increased strength of those bonds will improve the wear resistance of the sintered body and increase its life span. Diamond grains are added to a mixture of CBN grains, titanium grains, aluminum grains, and aluminum nitride grains. The function of diamond is several fold. First it reacts with the oxygen bound to the CBN resulting in carbon monoxide (CO) and a carbon bound to the CBN. The sintering process is performed after the mixture is exposed to high temperature in vacuum

conditions and the CO is pulled away from the mixture. The pulling away of CO and other volatile vapors from the preform also helps prevent pockets of gas from forming later during sintering and helps prevent further oxidation. Another function of diamond is that it reacts in situ with the titanium in the mixture to form titanium carbide, as well as reducing aluminum III oxide (Al_2O_3) to aluminum and titanium dioxide (TiO_2) to titanium. A third function of the diamond relates to the speed of the in situ chemical reactions. Since the CO is pulled away from the mixture, the concentration of oxides bound to CBN, Ti, or Al changes, and a shift in equilibrium appears. This drives the reaction in the direction of reducing the CBN grains, titanium grains, or aluminum grains and speeds up the reaction.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a top view of the CBN sintered body with a visible cobalt attachment ring.

FIG. 2 is a cross sectional view of the CBN sintered body with a cobalt infiltrated attachment ring.

DETAILED DESCRIPTION OF THE INVENTION

One of the concerns of present CBN sintered bodies is if there is sufficient attachment of the sintered body to its substrate. Our present invention focuses on trying to reduce that problem. We found by infiltrating cobalt into the CBN sintered body, primarily near the edges, from its substrate 11 that the strength of the attachment improved. We believe that a network of cobalt to cobalt bonds that extends from the adjacent substrate 11 into the CBN sintered body 15 is responsible for the improved attachment. In our preferred embodiment the infiltrated cobalt attachment ring 13 is formed in a CBN sintered body 15, using titanium carbide, titanium nitride, and

aluminum as metal binders in the CBN sintered body to encourage the attachment of CBN to CBN bonding. We believe that during the sintering process these metal binders act as catalysts to form those CBN to CBN bonds.

One of the unique results of our preferred embodiment is the exceptional hardness demonstrated by the CBN sintered body. Various mixtures of the weight percentages of the metal binders and various mixtures of the grains sizes of the metal binders and CBN grains were experimented with to obtain the high hardness. Our preferred embodiment and several other mixtures resulted in hardness ranging from 3500 Vicker's Hardness Units to 3900 Vicker's Hardness Units. The industry believes a competitive CBN sintered body will demonstrate at least 2900 Vicker's Hardness Units, so our preferred embodiment surpasses that requirement of the industry's approval.

In our preferred embodiment we use tungsten carbide as our substrate 11 which comprises 6% cobalt. During sintering the cobalt melts and infiltrates into the adjacent CBN sintered body 15 and the cobalt attachment ring 13 is formed. In the art, tungsten carbide with a concentration of cobalt is preferred for several reasons. Two of those reasons are because tungsten carbide demonstrates high hardness and tungsten carbide also seems to improve the thermal conductivity of the CBN sintered body. In addition to thermal conductivity, tungsten has a thermal expansion that is close to CBN, which reduces the stress between the two materials under high temperature.

Different weight percents of CBN grains in the CBN sintered body can serve different functions. In the art, a high CBN concentrated sintered body is used to cut materials like cast iron, while lower CBN concentrated sintered bodies are used to cut material like steel. Our preferred embodiment of a high CBN concentrated sintered body

comprises a 95 weight percent of CBN grains, but because it is hard to obtain exact weight percents of some of the grains, due to ball milling, the CBN grains usually fall in a range of 93 to 97 weight percent. However, we have had some success with a high CBN concentrated sintered body where the CBN grains were 90 to 93 weight percent. Our low CBN concentrated sintered body ranges from 50 to 70 weight percent of CBN grains.

The size of the CBN grains is also important because it affects the hardness and surface roughness of the CBN sintered body. Smaller grains also increase the total surface area of all the grains in the CBN sintered body's microstructure, which is believed in the art to improve CBN to CBN bonding, due to more opportunities for binding. In our preferred embodiment, the CBN grains are 1 to 3 microns. We believe that this reduces the surface roughness of the CBN sintered body. Reduced surface roughness also reduces the friction that is created while cutting hard materials, which is beneficial because less energy is wasted during cutting and the life of a motor, that could be used to operate a cutter, would increase. Another benefit of reduced friction is less heat is produced during cutting. A major concern for CBN sintered bodies is that thermal expansion of the metal binders in the CBN sintered body is greater than the thermal expansion of the CBN. This causes internal stress which cracks the CBN sintered body. A reduction in heat due to a reduction in friction during cutting will reduce the amount of thermal expansion within the CBN sintered body and prevent against cracking.

In one of our embodiments of our invention 60% of the CBN grains were 4 to 8 microns, 30% of the CBN grains were 12 to 22 microns, and 10% of the CBN grains were 1 to 2 microns. While the hardness of the CBN sintered body increased so did the

brittleness. Despite the increase in brittleness, the CBN sintered body worked well in tests and we intend for it to be protected in this patent.

While infiltrated cobalt is found throughout the entire CBN sintered body **15** a much higher concentration is found near the edges of the sintered body. Pictures from a scanning electron microscope (FIG. 1) reveal that the thickness of the attachment ring **13** is 15 to 30 microns. In our preferred embodiment, from a two dimensional view, our CBN sintered body is shaped like a triangle with rounded corners **14**. Around these corners **14** the thickness of the attachment ring **13** increases, where the thinner portions of the attachment ring are found along straight edges. While our preferred embodiment is shaped like a triangle with rounded corners from a two dimensional view, we don't intend to patent that design, but intend to patent any CBN sintered body with an attachment ring of infiltrated cobalt of any shape according our claims. The infiltrated cobalt attachment ring **13** is found along the entire interface **12** of the CBN sintered body **15** and its tungsten carbide substrate **11**.

Titanium carbide, titanium nitride and aluminum are metal binders found with in the CBN sintered body **15**. Aluminum is used in making CBN sintered bodies because of its reducing ability. Aluminum is usually found in its oxide form, Al_2O_3 . By adding aluminum in the preform, during sintering the aluminum removes oxygen molecules from the boron nitride surface as well as the surface from other metal binders such as titanium. We use aluminum as a reducing agent, although diamond is our primary reducing agent. Titanium carbide and titanium nitride are also well used in the art of fabricating CBN sintered bodies.

This invention provides a sintered CBN body with improvements over the current CBN sintered bodies on the market. Sintered CBN bodies are subjected to tasks exposing them to high amounts of heat, stress, and pressure. The current invention reduces the oxides that inhibit intercrystalline bonding during the sintering process and adds increased bond strength through more exposed surface areas that can form bonds.

This invention uses a unique process by allowing chemical reactions to happen in situ. In the preferred embodiment carbon, aluminum nitride, and CBN grains comprise a mixture that is mixed with submicron sized grains of titanium and aluminum. Before the sintering process these molecules will react under the heat and vacuum to become titanium nitride, titanium carbide, aluminum, and cubic boron nitride and form a preform. The preferred form of the carbon is diamond, while this may not be required for the process to work, graphite may work as well. In our preparation of the mixture, we try to have all of the grains be under 3 microns in size, where the range for CBN grains is 1 to 3 microns, the range for AlN grains is .3 to .5 microns, and the range for the carbon grains is .5 to 2.5 microns. The range for aluminum and titanium grains is hard to determine because they are obtained through milling, although we believe that they are submicron sizes. While the size of the grain is attempted to be smaller than 3 microns usually 3 to 4 percent of our volume often turns out to be larger than 3 microns.

Some believe that to maximize the strength of a CBN sintered body, the grain sizes should vary. The CBN molecules within larger CBN grains are already bound tightly together. The smaller grain sizes are intended to lubricate the larger grains and help all the grains fit compactly in the form of the preferred sintered body. The smaller grains can also fill the in wholes that the larger grains create. We believe; however, that

there is some benefit achieved by smaller grains, because increased surface area from the smaller grains provide for more and stronger bonds.

In the preferred embodiment the binding material comprising of titanium, aluminum, carbon (in the form of diamond), and aluminum nitride grains will only be about 5 weight percent of the mixture, where AlN grains are about 1 weight percent, carbon grains are about 2 weight percent, aluminum grains are about 1 weight percent, and titanium grains are about 1 weight percent. These are only target numbers, as it is hard to obtain the exact amounts. The remaining 95 weight percent is targeted to be CBN grains. Since it is difficult to obtain exact amounts, acceptable ranges are as follows: the range of CBN grains are 93 to 97 weight percent, the range of aluminum nitride grains are .5 to 1.5 weight percent, the range of aluminum grains are .5 to 1.5 weight percent, the range of titanium grains are .5 to 1.5 weight percent, and the range of carbon grains are 1.5 to 2.5 weight percent. Since some of the carbon will react with oxides on the CBN grains and grains of the metal binders in the mixture and leave in the form of CO, the weight percent of carbon after sintering will be less than it was before reacting the compounds.

Boron cubic nitride, by itself, is inert. Due to the high amount of oxides that bind to the CBN grains, it is hard to create bonds between the CBN grains. Certain metals have shown to act as catalysts and induce the formation of intercrystalline bonds between the CBN grains. Aluminum, titanium, and silicon are among the some effective metals. In our preferred embodiment titanium combines with aluminum and nitrogen to form TiN and TiC, which act as our main binders. However, some aluminum also functions as a binder. It is unknown whether any AlN, pure carbon, pure titanium or pure nitrogen will

be present in the sintered body, although it is believed that they react completely. However, if any remain in the sintered body, we believe that they may also function as binders in the sintered body.

The titanium and aluminum are obtained by ball milling. The powders of diamond, CBN, and AlN are placed into an aluminum sleeve with titanium balls weighing about 250 grams. Acetone is used to enhance the mixing process. Acetone inhibits the grains from reacting with the oxygen in the air, and also separates the particles away from each other. While acetone is useful in the mixing process, it is not required. Certain alcohols can also be used to aid in mixing, such as isopropanol. Pressure has also been found useful during the milling process, but in our preferred embodiment we mill at atmosphere. During the milling process, submicron sized titanium grains break off from the balls and submicron sized aluminum grains break off from the aluminum sleeve. In order to obtain the weight percent of the retrieved aluminum and titanium, the titanium balls and the aluminum sleeve are weighted before and after the milling procedure. The difference between the weights identifies the amount of aluminum and titanium obtained. The submicron size of the aluminum and titanium is important because the smaller the grain sizes of the binders in the CBN, the more surface area there is to bind, which increases the amount of bonds forming during the sintering process. Further, one way that CBN sintered bodies break, is through the thermal expansion of the binders in the CBN. When the binders expand, they create a pressure on the CBN from the inside, which creates a stress that usually causes the first cracks in a CBN sintered body. However, when the grain sizes of the binders are small, the grains have less ability to expand. Each element has a coefficient of thermal

expansion, which describes how much that element will expand under a certain amount of heat. The equation for thermal expansion is $\Delta L = \alpha (\Delta T) (L_0)$, where α = the coefficient of thermal expansion, L = length, T = temperature, and L_0 = initial length.

For example, let's assume that particle A had a thermal expansion coefficient of .1. If particle A is 3 microns in size, then under a 200 C degree increase the grain size will expand to 60 microns. Then suppose that particle A is only 1 micron, and under the same conditions, particle A will only expand to 20 microns. This illustrates that the smaller grain sizes would expand to a smaller volume under heat. It is believed that this will improve the wear resistance of the CBN sintered body. By providing CBN, diamond, and AlN grains in the mixture in the aluminum sleeve with the titanium balls, the powders are distributed thoroughly in the mixture. Further, diamond and CBN are substantially harder than the aluminum and titanium. The hardness of the diamond and CBN grains help to wear down the titanium balls and aluminum sleeve.

After the milling process, the CBN mixture is heated to 700 degrees C. It is believed that this oven cycle will react titanium and aluminum nitride to form aluminum and titanium nitride. Other chemical reactions can occur during the oven cycle, but its main purpose is to react these two chemicals. During the oven cycle, carbon may begin reducing the oxides on the CBN grains, but this is not known. Too high of a temperature during the oven cycle is not advisable because too high of temperatures would could begin the sintering process. Since the mixture is not backed on its substrate and is without form, sintering in the oven stage would be counter productive. This oven cycle may not be necessary in this process of making a CBN sintered body, but is believed to help purify the sintering agents before actually sintering the preform. The reaction of

titanium and aluminum nitride to aluminum and titanium nitride could also be accomplished during a later stage. It is believed that the new chemistry of this mixture will aid in this process and result in a stronger CBN sintered body.

The mixture formed after the oven cycle is compacted into a Nb container. A tungsten carbide substrate, with 6 percent cobalt is pressed adjacent to the powder. The grooves in the tungsten carbide substrate and the Nb container provide the shape of the sintered body. The tungsten carbide functions as a backing substrate for the CBN sintered body. Before sintering, the mixture is exposed to an environment of heat in vacuum. The vacuum removes the gases and voids that are present in the mixture, preferably the vacuum is 10^{-5} torr to 10^{-6} torr. Usually 40% of the mixture has some kind of space present. It is important to remove these voids to avoid air pockets in the CBN sintered body. The heat in vacuum is hot enough to melt the copper cap, which seals off the Nb container and excludes other oxides and gases from entering which could interfere with the sintering process.

The carbon (in the diamond form) in the mixture reacts with oxides that bind to the surface of the CBN grains before the copper cap melts. The result is CO and an open bonding site for other CBN grains. The CO is pulled away by vacuum. The removal of CO will disrupt the equilibrium and drive the reaction towards a new equilibrium with more products, which is the CO. As more CO is made, it is also removed. This process speeds up the chemical reactions. It is important that gases, such as CO, are removed from the preform. In this method, this is accomplished by vacuum. Gases, such as CO, have the potential of reoxidizing the CBN grains. Because all chemical reactions must maintain a state of equilibrium, if the CO were present then some of the oxides would not

react with the carbon, leaving potential CBN to CBN bonding sites occupied with an oxide. The removal of the oxides does increase the amount of the CBN intercrystalline bonds. In addition to carbon reducing the CBN grains, carbon also reduces with titanium grains and aluminum grains. The following reactions are described as; $\text{Al}_2\text{O}_3 + 3\text{C} \rightarrow 2\text{Al} + 3\text{CO}$ and $\text{TiO}_2 + 3\text{C} \rightarrow \text{TiC} + 2\text{CO}$. The net results of heating in vacuum are fewer oxides to inhibit binding and the chemical reactions to form aluminum and TiC which act as binders in the sintered body. This heating in vacuum stage creates a preform for sintering. Again the removal of CO drives the chemical reactions to form more TiC and aluminum. Most of the carbon is used as either a reducing agent that leaves during the heating in vacuum stage in the form of CO or that it combines with the titanium to make TiC. We believe that some carbon could be leftover in the preform, which would also be present in the final sintered body. We believe that the remaining carbon will react with small amounts of niobium from the niobium container to form niobium carbide and tungsten from the substrate to form more tungsten carbide.

The sealed, Nb container is placed into a high temperature/ high pressure chamber. In our preferred embodiment this chamber will be a salt/pyro cell. Salt/pyro cells are well established in the art, providing high temperature and high pressure. The preform adjacent to the tungsten carbide in the sealed Nb container is sintered at roughly at 1400 degrees C and at a pressure of 55 kbars. The strength of a sintered body is gained by reducing the surface areas of the grains by compacting the grains through heat and pressure. During the sintering process the grains move closer together and the areas between the grains become much smaller. The edges of the grains mash up next to each other and form intercrystalline bonds. The surface energies of the individual grains are

converted into the bond energies. The more surface area that a grain has the more bonds it will be able to make. We believe that by reducing the size of the grains that we can increase the bonding energies and the amount of bonds in the sintered body.

The cobalt in the tungsten carbide melts during the sintering process and distributes itself throughout the sintered body, but is mostly found near the edges of the sintered body. It is not known why the cobalt has not distributed itself throughout the entire CBN sintered body evenly, but the high concentration of infiltrated cobalt does give added strength to the attachment of the substrate to the CBN sintered body.

After the sintering process is complete, the temperature is removed and then the pressure is also removed and a unique sintered body is recovered. Due to less oxides bound to the CBN grains, titanium grains, and aluminum grains during the sintering process, this sintered body has increased wear resistance and greater bond strength.